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USE OF ACCELERATED HELIUM-3 IONS FOR DETERMINING
OXYGEN AND CARBON IMPURITIES IN SOME PURE MATERIALS

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16. Abstract Methods are developed for the determination of O impurity in Be and Si carbide and concurrent determination of C and O impurities in Si and W by irradiation with accelerated He-3 ions and subsequent activity measurements of C-11 and F-18 formed from C and O with the aid of a gamma-gamma coincidence spectrometer. Techniques for determining O in Ge and Ga arsenide with radiochemical separation of F-18 are also described.			
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The present paper deals with the use of ^3He ions for the /181* determination of oxygen and carbon impurities in beryllium, silicon carbide, silicon, gallium arsenide and tungsten. The impurities to be determined were recorded with a γ, γ -coincidence spectrometer. This instrument records the activity of the radioisotopes ^{11}C (β^+ , $T_{1/2} = 20.4$ min) and ^{18}F (β^+ , $T_{1/2} = 110$ min) which are formed by the capture of ^3He ions by carbon and oxygen nuclei [1,2].

Examination of the nuclear reactions and γ -spectra of the irradiated specimens showed that irradiation of some substances leads to the formation of large quantities of radioactive isotopes which interfere with the oxygen and carbon determination. In our studies, germanium and compounds of the type A_3B_5 were found to be strongly-activating substances of this kind. To analyze such substances, it was necessary to include in our analytical procedure the radiochemical purification of the isotope to be determined. The direct instrumental method can be employed if either no positron-active isotopes form during ^3He irradiation, or their half-life is much shorter than the half-life of the isotope to be recorded [1-6]. The direct instrumental method is suitable for determining the oxygen content of beryllium and silicon carbide, and for the concurrent determination of oxygen and carbon in silicon and tungsten.

The specimens were irradiated in the cyclotron of the

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I. V. Kurchatov Institute of Atomic Energy with a 13-MeV ^3He ion beam. The interchange of specimens was accomplished in one minute with the aid of an automatic device. After irradiation, the specimens were cooled in running water. The beam current and irradiation time were selected in accordance with the physical properties and purity of the material to be studied. Optical quartz, silicon carbide and tungsten carbide were used as standards.

A characteristic feature of this method is its high sensitivity at relatively small depths of activation. An energy of 13 MeV corresponds to an ion path of $75\mu\text{m}$ in gallium arsenide and $35\mu\text{m}$ in tungsten. A portion of this path is taken up in the surface film, whose oxygen content may greatly exceed that of the bulk of the specimen. The presence of a film on the beryllium and silicon carbide specimens was the reason that the oxygen contents of 0.3% found for beryllium and 0.04% for silicon carbide are relatively high and vary little among technologically-different specimens. It was shown in [5] that the effect of a film on silicon and germanium specimens can be eliminated by removing the surface layer to a depth of $20\mu\text{m}$ if the specimens are chemically polished the day before irradiation. Analogous results were obtained for gallium arsenide specimens. /182

Because the ^3He paths are much shorter in the heavy metals, surface treatment assumes a special significance for these specimens. Before irradiation the specimens were carefully polished with powders and treated with a hot acid mixture ($\text{HF}:\text{HNO}_3 = 1:1$) until a mirror finish was obtained. The necessary etching depth was estimated through layer-by-layer etching of the specimen after irradiation. The function obtained, relating ^{18}F activity to the tungsten etching depth, indicates that an etching depth of $5\text{-}6\mu\text{m}$ is needed to remove the ^{18}F

which forms in the surface layer.

The specimens to be analyzed were irradiated by a 13-MeV beam of ^3He ions. The ions lost a total energy of 3 MeV (from 13 to 10 MeV) while traversing the surface oxide film. Tungsten specimens were etched to a depth of $12\mu\text{m}$; the remaining active layer was used in determining the oxygen and carbon content of the bulk phase of the tungsten. The procedure for determining carbon and oxygen in the tungsten, as well as silicon [5], was as follows. The sample to be analyzed was irradiated with ^3He ions (energy = 13 MeV, beam current = $3\mu\text{A}$) for one hour, and then its surface layer was removed to a depth of $12\mu\text{m}$ by etching in a 1:1 mixture of nitric and hydrofluoric acid. The fall in the activity of the sample was then measured with a γ,γ -coincidence spectrometer. ^{11}C positron activity was generally observed against a background of ^{18}F activity; hence the sensitivity of the carbon determination depended on the oxygen concentration in the sample.

The carbon and oxygen contents found in the tungsten specimens were 10^{-4} and $10^{-5}\%$, respectively, at a sensitivity of $n \cdot 10^{-7}\%$. The carbon content of the silicon specimens ranged from 10^{-4} to $10^{-6}\%$ according to measurements.

To determine the oxygen content of strongly-activating substances, it was necessary to employ chemical methods of separating and purifying the fluorine. Two methods were proposed in [5] for the deep purification of fluorine to remove arsenic, selenium and germanium isotopes which form from germanium on irradiation with ^3He ions. The first method is based on the distillation of fluorine in the form of fluosilicic acid in the presence of perchloric acid in a water vapor stream at $120-140^\circ$. When we used this method, considerable

quantities of arsenic and selenium entered the distillate. We therefore included the preliminary steps of precipitating silver arsenate and selenate and lanthanum fluoride. This procedure ensures a high fluorine purification coefficient, but is time-consuming and laborious. The second method is based on the precipitation of lanthanum fluoride and the removal of arsenic and selenium impurities by calcinating the lanthanum fluoride in a stream of inert gas and hydrogen at 1100°C. This technique is less laborious but is suitable only for the separation of volatile impurities. /183

In our effort to determine small contents of oxygen (10^{-5} - $10^{-6}\%$) in various substances, especially compounds of the type A_3B_5 , we focused our attention on perfecting the technique of fluorine purification. To this end, we studied the behavior of fluorine when added to a solution of crushed quartz and sulfuric acid ($HF:HNO_3:H_2O = 1:2:4$) without additional heating. We found that under these conditions, 75% of the fluorine is distilled off in an air stream in 15 minutes. The separation of arsenic, selenium and gallium gave a fluorine purification coefficient on the order of 10^7 . This technique was employed in the analysis of germanium and gallium arsenide.

In the case of germanium, a single fluorine-distillation operation followed by the precipitation of lanthanum fluoride was sufficient to obtain a radiochemically pure fluorine. When gallium arsenide was analyzed, a step was added: the precipitation of silver bromide to remove the bromine formed in the fluorine by interaction of the 3He ions with the arsenic. Owing to this technique, purification of the fluorine was reduced to the following procedure: The layer of irradiated material to be analyzed is dissolved in 5 ml of a hydrofluoric acid-nitric acid mixture; this is transferred to a glass distillation flask containing 1 gram of finely-crushed quartz. The flask is covered, and 10 ml of sulfuric acid is added from

a dropper over a 2-3 minute period. The flask is connected via a system of washing and absorption vessels to a water-jet pump for passing an air stream through the reacting mixture. The silicon tetrafluoride which is separated is absorbed by water; the fluorine is precipitated in the form of lanthanum fluoride, and its activity is measured. The time required for the separation of radiochemically pure fluorine is 20 minutes for germanium and 30 minutes for gallium arsenide.

The technique described was used to analyze technological specimens of gallium arsenide. The results showed that some samples of gallium arsenide have an oxygen content of $(1-3) \cdot 10^{16}$ atoms/cm³ with an error of $\pm 40\%$; the oxygen content of their films (500 μ m) was found to be $(5 \pm 2) \cdot 10^{17}$ atoms/cm³.

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